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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to the porosity silicon carbide fiber structure which can be used as heat-resistant filters, such as an engine abandonment gas filter, and can be applied to the insulator of elevated-temperature objects, such as an engine and a combustion furnace, etc.

[0002]

[Description of the Prior Art]Use of silicon carbide is studied from the former as porous ceramics which can be used as the filter and insulator which bear high temperature environment. For example, after mixing other raw materials to silicon carbide powder and fabricating to Plastic solids, such as honeycomb shape, the art calcinated at an elevated temperature is indicated by JP,7-172952,A and JP,10-15323,A.

[0003]However, it is necessary to calcinate a big structure repeatedly, and there is a problem in respect of calling it productivity in such art. Since the matrix of ceramics itself is not porosity, it is necessary to form opening structure like a honeycomb. It is thought by using Webb of the porosity formed from silicon carbide fiber for the above-mentioned material from such a viewpoint that the two above mentioned faults are cancelable. When a porous-structure object is formed from textiles, there is also an advantage of being easy to manufacture what of small fine pores is [ heat-resistant impact strength with strong intensity ] strong in addition to this.

[0004]This invention persons produce previously the two dimensions or the three-dimensional structure object in which specific surface area consists of fibrous activated carbon of 100-2500 m<sup>2</sup>/g in JP,9-118566,A, Subsequently, the fibrous activated carbon and silicon monoxide gas which constitute this structure are made to react at the temperature of 800 \*\* - 2000 \*\*, After making the two-dimensional or three-dimensional structure which consists of silicon carbide fiber obtained contain an organosilicon compound, The method of obtaining a silicon carbide

fiber structure by drying, and heating in temperature of 800-2000 \*\* continuously in the vacuum below  $10^2$ Pa or the gas atmosphere which does not contain oxygen substantially was proposed. However, since a structure is formed before silicon-izing this method or textiles, there is a problem of the dimensional change by a reaction, and it cannot be said to be thing sufficient in a strong field. Similarly the manufacturing method of the silicon carbide fiber used for this art is based on the art given in JP,6-192917,A which this invention persons proposed previously.

[0005]

[Problem(s) to be Solved by the Invention]Then, this invention makes it a technical problem to propose the manufacturing method of the porosity silicon carbide fiber structure which was excellent in heat resistance, intensity, and the size fitness of a structure, and was excellent also in productivity.

[0006]

[Means for Solving the Problem]In order to solve said technical problem, this invention adopts the following composition. "Namely, the 1st invention of this invention, density makes a binding material exist between textiles of Webb who consists of silicon carbide fiber which is  $3.0-3.2\text{g/cm}^3$ , It is a manufacturing method of a porosity silicon carbide fiber structure whose apparent density gravity is  $0.05-1.0\text{g/cm}^3$  making this silicon carbide fiber and this binding material sinter under decompression or an inert gas atmosphere."

[0007]"The 2nd invention of this invention, an oxygen content makes a binding material exist between textiles of Webb who consists of silicon carbide fiber which is 1 or less % of the weight, It is a manufacturing method of a porosity silicon carbide fiber structure whose apparent density gravity is  $0.05-1.0\text{g/cm}^3$  making this silicon carbide fiber and this binding material sinter under decompression or an inert gas atmosphere."

[0008]An invention of the 3rd of this invention is a manufacturing method of a silicon carbide fiber structure, wherein these silicon carbide fiber is the textiles manufactured at a process of following (A) - (B) in said 1st or 2nd invention.

Specific surface area according [ a fiber diameter ] to a BET nitrogen absorption method at 1-20 micrometers (A) An activated carbon fiber of  $700-1500\text{ m}^2/\text{g}$ , A process to which at least one sort of gas chosen from silicon and a silicon oxide is made to react under 1200-1500 \*\* temperature conditions under decompression or an inert gas atmosphere. (B) A process of heat-treating textiles obtained at said process at 1700 \*\* - 2100 \*\* the bottom of existence of a boron compound, and in an inert gas atmosphere.

[0009]An invention of the 4th of this invention is a manufacturing method of a silicon carbide fiber structure by which at least one sort as which a binding material is chosen from silicon carbide, alumina, silica, zirconia, and a titania being included as an ingredient in said 1st [ the ]

- the 3rd one of inventions.

[0010]As a result of examining said technical problem, first, this invention persons manufactured silicon carbide fiber, they formed a structure for these textiles continuously, and found out that a method of sintering with a binding material was preferred.

[0011]Although it is not a porous body in a place, to JP,6-179755,A. Silicon carbide system staple fiber preforming to which a silicon carbide system staple fiber is joined with a carbosilane system polymer is described, A method of mineralizing a carbosilane system polymer which heated this preforming to temperature of the range of 200-1300 \*\* in an inert gas atmosphere, and has joined a silicon carbide system staple fiber is indicated. To JP,8-22782,B, a ceramic staple fiber and ceramic powder A drainage system, After mixing in a fluid of a non-drainage system, or fluids, such as a melting wax and melting resin, and fabricating this in a mold etc. in predetermined shape, make it dry, form a Plastic solid and within a vacuum atmosphere, After removing air in an opening in a Plastic solid, impregnate the Plastic solid with a liquefied ceramics precursor with gas pressure etc., and heat a Plastic solid and the above-mentioned ceramics precursor is made to convert into ceramics, After repeating this being impregnated and heating several times, it is said that this Plastic solid is sintered under an elevated temperature and high voltage, such as isostatic press between heat, and a Plastic solid is made to elaborate about how to obtain fiber-reinforced ceramics.

[0012]although applied such art, Webb who becomes said JP,6-192917,A from silicon carbide fiber of a statement or "NIKARON" textiles currently used by said JP,6-179755,A, "tee llano" textiles, etc. were used, and Webb was formed and being calcinated with a binding material, Intensity of a structure was not enough and a dimensional change was looked at by calcination. Then, as a result of inquiring wholeheartedly further that these faults should be improved, it discovers that there is a problem about the textiles themselves which forms Webb, and completion of this invention is reached.

[0013]

[Embodiment of the Invention]Form Webb from silicon carbide fiber, a binding material is made to exist between the textiles of this WEBBU, and it is made to sinter at an elevated temperature in this invention. Here, a nonwoven fabric or textiles like felt are the sheet shaped thing which gathered, and Webb is not in the state which textiles combined firmly with the binding material. Webb may be two-dimensional sheets, such as circular and a quadrangle, and honeycomb structure, wave shape, core box shape, etc. may be fabricated in three dimensions. It may be wound like a roll.

[0014]In this invention, the silicon carbide fiber which forms said Webb needs to be whether density is  $3.0\text{--}3.2\text{g/cm}^3$  and 1 or less % of the weight of oxygen contents. If a size changes easily in less than 3.0 textiles at the time of sintering of a structure and a binding material in density and an oxygen content increases exceeding 1 % of the weight, the weight loss by

oxidation is produced at the time of sintering, and it is not desirable in respect of a dimensional change and intensity.

[0015]Although the silicon carbide fiber which density is  $3.0\text{--}3.2\text{g/cm}^3$ , or is 1 % of the weight of oxygen contents is obtained also by the method of heat-treating general silicon carbide fiber with a sintering aid, It is most preferred to manufacture textiles by the method explained below in respect of the productivity of textiles and the intensity of the textiles themselves.

[0016]The silicon carbide fiber which was most suitable for <manufacturing method of desirable silicon carbide fiber> this invention, It is obtained by heating further the silicon carbide fiber manufactured by making an activated carbon fiber, and silicon and a silicon oxide react (A process may be called below) at  $1700^{\circ}\text{C}$  -  $2100^{\circ}\text{C}$  the bottom of decompression, or in an inert gas atmosphere under existence of a boron compound. This process may be called B process below. In addition to a boron compound, in B process, carbon compounds may exist further.

[0017]Hereafter, it explains in detail from A process first. It is indicated by the patent No. 2663819 gazette about the manufacturing method of the textiles to which silicon and the silicon oxide, and the activated carbon fiber were made to react. That is, a fiber diameter can use the method to which specific surface area makes the porous carbon textiles and gaseous silicon monoxide of  $100\text{--}2500\text{ m}^2/\text{g}$  react at  $800\text{--}2000^{\circ}\text{C}$  at  $5\text{--}100$  micrometers.

[0018]In said activated carbon fiber, a  $0.1\text{--}50\text{-mm}$ -long staple fiber and the continuous fibers (a filament, yarn, etc.) which do not have restriction of length substantially may be included, and these may be any, such as cotton yarn, monofilament yarn, and multifilament yarn. The activated carbon fiber may be formed in sheet like bodies (felt, cloth, etc.) or other three-dimensional structure objects (a honeycomb, a pipe, a three-dimensional weaving thing, etc.).

[0019]The specific surface area according [ said activated carbon fiber ] to a BET nitrogen absorption method is a thing of  $700\text{--}1500\text{ m}^2/\text{g}$ . Since carbon with specific surface area unreacted in the textiles obtained by a silicon-ized reaction fully not advancing in a silicon carbide-ized reaction process by less than  $700\text{ m}^2/\text{g}$  remains, the feature of silicon carbide fiber must fully have been revealed as a result. The intensity of the silicon-carbide-ized textiles which the intensity as an activated carbon fiber runs short, and yield [ in / when large / exceeding  $1500\text{ m}^2/\text{g}$  / an activated carbon-ized (activation) process ] is low and specific surface area is not only uneconomical, but are obtained as a result also becomes insufficient.

[0020]The activated carbon fiber used for the method of this invention of having the aforementioned characteristic is manufactured by a publicly known method. The activated carbon fiber which uses as a raw material the pitch obtained from a fossil fuel by JP,6-306710,A, for example about the method of activated-carbon-izing the precursor textiles which consist of organic substances is indicated. Especially when making the staple fiber of an

activated carbon fiber profitably like, this is staple-fiber-ized, after activated-carbon-izing said precursor textiles which spinning was beforehand carried out to the desired length, or were cut by the desired length or activated-carbon-izing precursor textiles.

[0021]In order to form an activated carbon fiber in the shape of sheet-shaped structures (a sheet, a web, etc.) beforehand, For example, the process which carries out melt spinning of the pitch and is made into continuation (merit) textiles as indicated by JP,2-255516,A, The method of performing continuously the process which is made caught and depositing these textiles and is made into a web, the process of carrying out non-deliquesce [ of this sheet-shaped fiber aggregate ], and the process of carrying out activation of the sheet which carried out non-deliquesce can be used. The precursor textiles for carbon fiber and carbon fiber can be formed in a felt-like sheet by dry type or a wet type, and the thing which carried out activated carbon fibrosis of this, or the thing which sheet-ized the staple of the activated carbon fiber can be used for this invention.

[0022]On the other hand, the thing which carried out corrugated processing and rolled a two-dimensional structure like the aforementioned felt or cloth, or the thing which processed honeycomb shape can be used for the three-dimensional structure object of an activated carbon fiber. A staple is made to adhere to a porous mold with a suction method, and a fiber structure object can also be acquired from the slurry which made water etc. distribute the staple of an activated carbon fiber thru/or its precursor.

[0023]As a silicon supply source containing at least one sort chosen from silicon and the silicon oxide which are used for A process, the powder mixture of silicon and a silica dioxide, the powder mixture of carbon and a silica dioxide, silicon monoxide, etc. can be mentioned. If an activated carbon fiber is heated at 1200-1500 \*\* with at least one sort of said silicon and a silicon oxide, the gas of silicon and/or a silicon oxide will be emitted, these will react to activated carbon, and an activated carbon fiber will be changed into silicon carbide fiber.

[0024]Next, B process explains. The bottom of decompression, or in an inert gas atmosphere, the silicon carbide fiber used suitably for this invention heat-treats at 1700 \*\* - 2100 \*\* under existence of a boron compound to the silicon carbide fiber manufactured at the above-mentioned A process. As a boron compound, simple substance boron, boron carbide, boric acid, etc. are used suitably. There is a method which makes it about 0.1-10-micrometer powder in the case of insoluble solids, such as simple substance boron and boron carbide, and textiles are made to support as a method of making it coexist with the silicon carbide fiber which should heat-treat these. In this case, since powder is fixed to textiles, using a suitable binder does not interfere, either. In the case of soluble boron compounds, such as boric acid, it can melt in suitable solvents, such as water, it can be used as a solution, and the method etc. with which this is impregnated in textiles can be used.

[0025]In this case, when weight of silicon carbide fiber to be heat-treated is made into 100

copies, the quantity of a suitable boron compound is converted into simple substance boron weight, and 0.1-10 copies are suitable for it. If there is less quantity of the boron made to live together than 0.1 copy, in order that sintering of the silicon carbide grain which constitutes textiles may not progress, what has the high intensity of textiles is not obtained and a fiber structure object with intensity high as a result is not acquired. If there is more quantity of boron than ten copies, the grain growth of silicon carbide grain will be controlled extremely, sintering will not progress, and textiles with intensity too high as a result will not be obtained. In addition to a boron compound, it can heat-treat to silicon carbide fiber if needed under existence of carbon compounds. As a publicly known method of adding these carbon compounds, High molecular compounds which make silicon carbide fiber to be heat-treated support impalpable powder-like carbon like carbon black, such as a method, phenol resin, and furan resin, are melted in a suitable solvent, this is impregnated in textiles to be heat-treated, and there is a method of carbonizing resin in atmosphere without oxygen. In this case, it is desirable to convert into carbon and to use carbon of about 0.1 to 2.0 times [ of the quantity of boron ] weight as a quantity of suitable carbon compounds.

[0026]Argon, helium, nitrogen, etc. can be used as an inert gas atmosphere in the case of heat treatment. 1700-2100 \*\* is preferred for the temperature of heat treatment, and 1900-2000 \*\* is still more preferred for it. When heat treatment temperature is less than 1700, since decomposition of silicon carbide fiber will become remarkable and the intensity of textiles will fall if long very sufficient effect is not acquired in heat treating time and it exceeds 2100 \*\*, it is not desirable. As for the temperature falling speed at the time of finishing the heating rate of the process in which it reaches to such heat treatment temperature, or heat treatment, and cooling, the amount of 50-100 \*\*/is suitable.

[0027]Although density is  $3.0\text{-}3.2\text{g/cm}^3$  or an oxygen content is the silicon carbide fiber which is 1 or less % of the weight, the silicon carbide fiber used for this invention, According to the aforementioned manufacturing method, density is  $3.0\text{-}3.2\text{g/cm}^3$ , and since an oxygen content will be 1 or less % of the weight, it is the most preferred.

[0028]<Shaping of a structure>, next shaping of a structure are explained. When silicon carbide fiber has already made a two-dimensional structure like felt, or the three-dimensional structure object, it can progress to the process which combines the textiles which constitute the structure promptly, but when silicon carbide fiber is a staple fiber, the process of fabricating this to a structure is required.

[0029]As a method of fabricating to a structure, a silicon carbide staple fiber can be performed using publicly known art. That is, suitable carrier fluid, such as water, can be made to be able to distribute a silicon carbide staple fiber as wet process, auxiliary materials, such as pulp, can be mixed if needed, and the structure of the two dimensions or three dimensions of desired shape can be made from deliquoring on a wire. It is the paper-making method for drying the

slurry in which the especially suitable method for this invention contains a silicon carbide staple fiber with wet process on an endless wire, and manufacturing the web of continuation. As a method of fabricating a silicon carbide staple fiber on a two-dimensional sheet, the dry method made to deposit until it drops in the air the textiles which carried out distributed floating with gravity in addition to the above-mentioned method and becomes desired thickness can also be used.

[0030]The textiles which constitute the silicon carbide fiber structure fabricated by these methods from this invention are combined with a heat-resistant binding material. As a heat-resistant binding material, alumina, silica, mullite, zirconia, The heat-resistant materials of oxide stock, such as a titania, silicon carbide and silicon nitride, boron carbide, The binding material which contains as an ingredient metal, such as high-melting point substances, such as a binding material which includes the heat-resistant materials of non-oxide stock, such as aluminimum nitride, as an ingredient or carbon, and boron, or titanium, nickel, and silicon, is mentioned. For example, it may use mixing carbon and/or silicon etc. for a silicon carbide binding material combining said binding material.

[0031]A publicly known method can be used as a method of adding these binding materials to a silicon carbide fiber structure. That is, a structure can be impregnated with what made suitable carrier fluid distribute the impalpable powder of material, and was made into the slurry, and the method of making impalpable powder sintering by calcinating this after desiccation, and making a binding action revealing can be used. In this case, choosing the suitable auxiliary agent which makes the above-mentioned slurry promote sintering, and adding is also performed. This method can be used to materials, such as alumina, silica, and mullite. This can be calcinated and a binding action can also be made to reveal, after impregnating a fiber structure object with the material which contains a metal alkoxide as an ingredient and making an alkoxide hydrolyze. The material which contains the alkoxide of silicon like a tetraethoxysilane, the alkoxide of aluminum like aluminum isopropoxide, or the alkoxide of titanium like titanium isopropoxide as an ingredient as an example of a metal alkoxide can be raised. It is possible to dissolve a ceramics-ized organic Si-polymer material in melting or a suitable solvent, and to also make said silicon carbide fiber structure impregnated by calcinating by an inert atmosphere. There are polycarbosilane and polysilazane in such an organic Si-polymer compound.

[0032]As a method of combining the textiles in a silicon carbide fiber structure by the silicon carbide which is the same material, a publicly known method can be used as a sintering process of silicon carbide. The example with the reaction sintering method which makes silicon carbide generate by the reaction of carbon and silicon. It is the method of impregnating a fiber structure object with the powder of a carbon source and silicon by a slurry, making carbon and silicon react because more than the melting point of silicon heats this from 1410 \*\* to 1600 \*\*

preferably in a vacuum or an inert gas atmosphere, making silicon carbide generate, and combining textiles. As for a carbon source, in this method, it is possible that they are also particles like carbon black and for it to be also an organic compound which generates carbon by heating in atmosphere without oxygen like phenol resin. In this method, silicon can also contact the structure containing only carbon to the silicon fused in a vacuum or an inert gas atmosphere, without impregnating with a fiber structure object.

[0033]In applying an ordinary pressure sintering process publicly known as a sintering process of silicon carbide to the above-mentioned fiber structure object, A fiber structure object can be impregnated in the slurry which mixed the sintering aid which consists of powder, and boron and carbon of silicon carbide, and the textiles in a structure can be combined by calcinating this at 1700-2100 °C among an inert atmosphere after desiccation. The quantity of desirable boron and carbon is 1 to 5% to both the weight of silicon carbide powder.

[0034]In a binding material which was described above, the powder of a heat-resistant material always suitable as aggregate can be added. That is, powder, such as silicon carbide, alumina, and silica, can be used as aggregate. 0.05 - 1.00 g/cm<sup>3</sup> is preferred for the apparent density gravity of the silicon carbide fiber structure of this invention, and 0.10 - 0.50 g/cm<sup>3</sup> is more preferred for it. It can ask for apparent density gravity by  $\frac{\text{weight (g)}}{\text{volume (cm}^3\text{)}}$  of a silicon carbide fiber structure by the volume (cm<sup>3</sup>) which a structure outside surface surrounds. If the apparent density gravity of a silicon carbide fiber structure is lower than 0.05 g/cm<sup>3</sup>, in uses, such as a filter, intensity is not enough, and if apparent density gravity is larger than 1.00 g/cm<sup>3</sup>, in uses, such as a filter, the permeability of a fluid worsens and is not remarkably preferred.

[0035]

[Example]Although the following example explains this invention concretely, of course, the range of this invention is not limited by the following example. the maximum stress at the time of carrying out by the compressive strength of the silicon carbide fiber structure in an example compressing a structure with a material-testing machine (trade name tensilon, made in Oriental Baldwin) -- the area of a compression zone -- \*\*\*\* -- it asked by things. Area which compresses a sample was made into 7.07cm<sup>2</sup>, and head speed was considered as a part for 1-mm/.

[0036]It asked for the density (true density) of the silicon carbide fiber in a <measurement of fiber density> example by the liquid substitution method indicated to JIS R 7601. That is, it measured, using 23 °C distilled water as immersion fluid.

[0037]Measurement of <measurement of oxygen content> oxygen content was measured by oxygen nitrogen analysis device TC-436 made from LECO.

[0038]Manufacture of <Example 1> (1) silicon carbide fiber : 6 mm of fiber length, specific



surface area  $1000\text{m}^2/\text{g}$ , And a pitch system activated carbon fiber (trade name: made in [ Osaka Gas Co., Ltd. ] Reno Beth A-10) with a fiber diameter of 13 micrometers, drying at  $120^\circ\text{C}$  among an air blasting dryer for 5 hours -- 20 g of these dry textiles -- silicon powder (the first class in a reagent.) The Wako Pure Chemical Industries, Ltd. make 60g and 130 g of silica dioxide powder (first class in a reagent, Wako Pure Chemical Industries, Ltd. make) were well mixed with the granular material fully mixed with the mortar, and the work tube made from mullite with an inside diameter of 64 mm installed in the tubular furnace was filled up with this mixture covering a length of 150 mm. Passing argon gas (purity 99.99 capacity %) by a part for 500-ml/in this work tube, temperature up was carried out over 3 hours from a room temperature to  $900^\circ\text{C}$ , and also temperature up was carried out over 1 hour to  $1350^\circ\text{C}$ , and it held at  $1350^\circ\text{C}$  for 4 hours. It cooled over 5 hours to the room temperature after that.

[0039]The mixture of these textiles and a granular material was picked out from the furnace, and water was made to distribute textiles and a granular material with a stirrer (trade name: an agitator, the product made by SHIMAZAKI) in 50 l. of water. By letting the sieve of a 149-micrometer eye hole pass for these dispersion liquid, textiles were collected to the plus sieve, and the stream washed textiles further. The textiles after washing were dried in a  $120^\circ\text{C}$  air blasting dryer for 5 hours. The weight of the collected textiles was 25g. The mean fiber length of these textiles was 5 mm, and the fiber diameter was 13 micrometers. Density was  $2.8\text{g}/\text{cm}^3$  and the oxygen content was 5.0%.

[0040](2) Densification of silicon carbide fiber : 0.6 g of boron powder (reagent: amorphous, Wako Pure Chemical Industries, Ltd. make) was mixed with 20 g of these textiles, adding a small amount of methanol, and textiles were made to support boron. These textiles are put into the Tammann electric furnace which offered a graphite heating tube 200 mm in inside diameter, Temperature up was carried out over 60 minutes from a room temperature to  $1000^\circ\text{C}$ , 2.0 l./Shunting argon gas (purity 99.99 capacity %), temperature up was carried out in 30 minutes from  $1000^\circ\text{C}$  more to  $2000^\circ\text{C}$ ,  $2000^\circ\text{C}$  was held for 10 minutes, and it cooled over 4 hours to the room temperature further. Density was  $3.15\text{g}/\text{cm}^3$  and the oxygen content was 0.1%.

[0041](3) formation [ of a structure ]: -- making 20 l. of water distribute these textiles -- further -- 2.0 g of synthetic pulps (trade name: SWP, Mitsui Chemicals, Inc. make) -- in addition, a sheet 160 mm in diameter was produced with the hand papermaking sheet molding apparatus. This sheet was dried in a  $150^\circ\text{C}$  air blasting dryer for 2 hours. this sheet -- silica -- sol (trade name: SNOWTEX OUP, the Nissan chemicals company make) -- it was immersed in the liquid which mixed 50 weight sections, alumina sol (trade name: alumina sol 100, Nissan chemicals company make) 170 weight section, and water 220 weight section, surplus liquid was sucked up on Buchner, and it was made to dry at  $105^\circ\text{C}$  for 6 hours Temperature up of this sheet was

carried out from a room temperature to 1000 °C among the atmosphere with the electric furnace in 4 hours, 1000 °C was maintained for 1 hour, and it cooled to the room temperature in 5 hours. Weight was 22 g and the appearance density of this sheet was 0.15g/cm<sup>3</sup>.

[0042]It covers with 5 g of massive silicon monoxide (made in \*\*\*\*\* Stix) into the box made from <Example 2> black lead, What cut out the felt (trade name: KURAKUTIBU felt FT-300, made in Kuraray Chemical) of eyes 200 g/m<sup>2</sup> which consists of an activated carbon fiber to which specific surface area uses phenol resin of 1000 m<sup>2</sup>/g as a raw material on it to 50x50 mm (weight of 0.50g) is carried, The lid made from black lead was put. Temperature up was carried out from a room temperature to 1350 °C in 4 hours, having put this into the tubular furnace provided with the work tube made from alumina 70 mm in inside diameter, and decompressing the inside of a work tube using the oil revolving vacuum pump of 50 l./s of exhausting capability. It was 0.72g, when were amounted to 1350 °C, and held the temperature for 2 hours, it cooled to the room temperature over 6 hours, felt was taken out and the weight was measured.

[0043]After being immersed in the dispersion liquid which made methanol distribute 1 g of boron (reagent: amorphous, Wako Pure Chemical Industries, Ltd. make), and set this felt to 100 g, this was taken out, the blotting paper was made to absorb excessive liquid and it was removed. It put into the Tammann electric furnace which put this felt on the board made from graphite, and was provided with a graphite heating tube 50 mm in inside diameter, temperature up was carried out in 30 minutes from a room temperature to 1000 °C, and temperature up was carried out in 30 minutes to 2000 more °C. When amounted to 2000 °C, the temperature was held for 10 minutes, and it cooled in 2 minutes to 1700 °C, it cooled to the room temperature further in 3 hours, and felt was taken out. This heating operation performed argon (99.9 capacity %) with a 2.0-l./m sink in the furnace. The weight of the felt after heating was 0.68g. As a result of taking out textiles and measuring density and an oxygen content, density was 3.1g/cm<sup>3</sup> and the oxygen content was 0.1%.

[0044]To this felt, SiC-powder 10 weight section, boron powder 0.002 weight section, It impregnated with the thing which made methanol 90 weight section distribute phenol resin 0.002 weight section (trade name TAMANORU 531, Arakawa Chemical Industries, Ltd. make), felt was carried on the wire gauze of 28 meshes, and superfluous liquid was removed. This felt was dried in a 105 °C air blasting dryer for 3 hours. It put into the Tammann electric furnace which put the felt after desiccation on the board made from graphite, and was provided with a graphite heating tube 50 mm in inside diameter, temperature up was carried out in 30 minutes from a room temperature to 1000 °C, and temperature up was carried out in 30 minutes to 2000 more °C. When amounted to 2000 °C, the temperature was held for 10 minutes, and it cooled in 2 minutes to 1700 °C, it cooled to the room temperature further in 3 hours, and felt was taken

out. This heating operation performed argon (99.9 capacity %) with a 2.0-l./m sink in the furnace. The weight of the felt after heating was 1.30g, and density was  $0.17\text{g/cm}^3$ .

[0045]in formation of the structure in (3) of <Example 3> example 1 -- the sheet (22g) of silicon carbide fiber -- silica -- instead of the mixed liquor of sol and alumina sol being impregnated, It impregnated with 120 g of things which dissolved phenol resin (trade name TAMANORU 531, Arakawa Chemical Industries, Ltd. make) in methanol by concentration 20% of the weight, and was made to dry with a 105 \*\* air blasting dryer for 3 hours. The weight of this sheet was 46g. Exhausting with an oil diffusion pump in the vacuum furnace of 300x300x300 mm of effective heating regions, temperature up of this sheet was carried out over 4 hours from a room temperature to 1450 \*\*, and it was held at that temperature for 1 hour. It cooled over 5 hours to the room temperature after that. The weight of the sheet at this time was 31g. 32-g silicon powder (reagent: 99%, 325 meshes, Aldrich make) was sprinkled over this sheet, and again, exhausting with an oil diffusion pump in a vacuum furnace, temperature up was carried out over 4 hours from a room temperature to 1450 \*\*, and it held at that temperature for 1 hour. It cooled over 5 hours to the room temperature after that. The weight of this sheet was 58g. The apparent density gravity of this sheet was  $0.41\text{g/cm}^3$ .

[0046]<Comparative example 1> (when not performing heat treatment under boron existence) 6 mm of fiber length, and specific surface area  $1000\text{m}^2/\text{g}$ , and a pitch system activated carbon fiber (trade name: made in [ Osaka Gas Co., Ltd. ] Reno Beth A-10) with a fiber diameter of 13 micrometers, drying at 120 \*\* among an air blasting dryer for 5 hours -- 20 g of these dry textiles -- silicon powder (the first class in a reagent.) The Wako Pure Chemical Industries, Ltd. make 60g and 130 g of silica dioxide powder (first class in a reagent, Wako Pure Chemical Industries, Ltd. make) were well mixed with the granular material fully mixed with the mortar, and the work tube made from mullite with an inside diameter of 64 mm installed in the tubular furnace was filled up with this mixture covering a length of 150 mm. Passing argon gas (purity 99.99 capacity %) by a part for 500-ml/in this work tube, temperature up was carried out over 3 hours from a room temperature to 900 \*\*, and also temperature up was carried out over 1 hour to 1350 \*\*, and it held at 1350 \*\* for 4 hours. It cooled over 5 hours to the room temperature after that. The mixture of these textiles and a granular material was picked out from the furnace, and water was made to distribute textiles and a granular material with a stirrer (trade name: an agitator, the product made by SHIMAZAKI) in 50 l. of water. By letting the sieve of a 149-micrometer eye hole pass for these dispersion liquid, textiles were collected to the plus sieve, and the stream washed textiles further. The textiles after washing were dried in a 120 \*\* air blasting dryer for 5 hours. The weight of the collected textiles was 25g. The mean fiber length of these textiles was 5 mm, and the fiber diameter was 13 micrometers. 20 l. of water was made to distribute 20 g of these textiles, 2.0 g of synthetic pulps (trade name: SWP, Mitsui

Chemicals, Inc. make) were added further, and a sheet 160 mm in diameter was produced with the hand papermaking sheet molding apparatus. This sheet was dried in a 150 °C air blasting dryer for 2 hours. The density of textiles was 2.8g/cm<sup>3</sup> and the oxygen content was 5.1%.

[0047]this sheet -- silica -- sol (trade name: SNOWTEX OUP, the Nissan chemicals company make) -- it was immersed in the liquid which mixed 50 weight sections, alumina sol (trade name: alumina sol 100, Nissan chemicals company make) 170 weight section, and water 220 weight section, surplus liquid was sucked up on Buchner, and it was made to dry at 105 °C for 6 hours Temperature up of this sheet was carried out from a room temperature to 1000 °C among the atmosphere with the electric furnace in 4 hours, 1000 °C was maintained for 1 hour, and it cooled to the room temperature in 5 hours. This sheet is [ weight ] 160 mm in diameter in 21 g and 7 mm in thickness.

The appearance density for which it asked from these was 0.15g/cm<sup>3</sup>.

[0048]<Comparative example 2> (when the density of a structure is low)

Instead of impregnating with 120 g of things which dissolved phenol resin in methanol by concentration 20% of the weight at the sheet (dry weight of 22g) of silicon carbide fiber in Example 3, It impregnated with 120 g of things which dissolved the same resin in methanol by concentration 10% of the weight, and was made to dry with a 105 °C air blasting dryer for 3 hours. The weight of this sheet was 34g. Exhausting with an oil diffusion pump in the vacuum furnace of 300x300x300 mm of effective heating regions, temperature up of this sheet was carried out over 4 hours from a room temperature to 1450 °C, and it was held at that temperature for 1 hour. It cooled over 5 hours to the room temperature after that. The weight of the sheet at this time was 26g. 14-g silicon powder (reagent: 99%, 325 meshes, Aldrich make) was sprinkled over this sheet, and again, exhausting with an oil diffusion pump in a vacuum furnace, temperature up was carried out over 4 hours from a room temperature to 1450 °C, and it held at that temperature for 1 hour. It cooled over 5 hours to the room temperature after that. The weight of this sheet was 38g. The apparent density gravity of this sheet was 0.04g/cm<sup>3</sup>.

[0049]The result of having measured the compressive strength of the silicon carbide fiber structure produced in the above example and comparative example was summarized in Table 1.

Table 1 Apparent density gravity and compressive strength of fiber structure object [0050]  
[Table 1]



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[0051]

[Effect of the Invention]According to Table 1, each silicon carbide fiber structure of this invention shows good intensity, but since [ that its density of the silicon carbide fiber before \*\*\*\* (ing) a structure is low and ] the structure produced in the comparative example 1 has the high oxygen content, its strong fall is remarkable. In the comparative example 2, since the quantity of a binding material of a suitable thing is insufficient, the density of a structure becomes smaller than 0.05, and the density and the oxygen content of silicon carbide fiber did not come to have sufficient intensity. The silicon carbide fiber structure of this invention can manufacture a structure with the difficult intensity with old art so that clearly from the above example.

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[Translation done.]